

DERIVATIVES OF PIPERAZINE AND MORPHOLINE. I.
ROUTING TO ALPHA, BETA-UNSATURATED KETONES

By
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A Doctoral Thesis to the Faculty Council of
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In Partial Fulfillment of the Requirements for the
Degree of Doctor of Philosophy



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TABLE OF CONTENTS

	Page
I. Scope of literature and introduction	i
II. Synthesis	12
III. Theoretical discussion	14
IV. Experimental details and procedures	
A. Addition products of piperazine	25
(1.) 1,4-dia-ethane-piperazine	25
(2.) 1,4-dia-(1-methyl-ethane)-piperazine	25
(3.) 1,4-dia-(1,1-dimethyl-ethane)-piperazine	27
(4.) 1,4-dia-(1,2-dimethyl-ethane)-piperazine	28
(5.) 1,4-dia-(1,3-dimethyl-ethane)-piperazine	31
(6.) 1,4-dia-(1,4-dimethyl-ethane)-piperazine	32
(7.) 1,4-dia-(1,4'-dimethyl-ethane)-piperazine	32
(8.) 1,4-dia-(1-methyl-2-ethyl-ethane)-piperazine	37
(9.) 1,4-dia-(1'-methyl-2-ethyl-ethane)-piperazine	38
(10.) 1,4-dia-(1-methyl-2'-methyl-ethane)-piperazine	41
(11.) 1,4-dia-(1,4'-dimethyl-2-ethyl-ethane)-piperazine	42
(12.) 1,4-dia-(1-methyl-2'-methyl-2-ethyl-ethane)-piperazine	45
(13.) 1,4-dia-(1-methyl-2'-ethyl-1,4'-ethane)-piperazine	47
(14.) 1,4-dia-(1-methyl-1,4'-ethane)-piperazine	47
(15.) 1,4-dia-(1-methyl-1,4'-dimethyl-ethane)-piperazine	51
(16.) 1,4-dia-(1-methyl-1,4'-dimethyl-2-ethyl-ethane)-piperazine	52
(17.) 1,4-dia-(1-methyl-1,4'-dimethyl-2-ethyl-ethane)-piperazine	52
(18.) 1,4-dia-(1-methyl-1,4'-dimethyl-2-ethyl-ethane)-piperazine	57

	Page																						
(20.) 1,4-bis-(2,4-dinitrophenyl)-2,5-dimethyl-3-bromo-benzene] <p style="padding-left: 40px;">polymerization</p> <tr> <td>(21.) 1,4-bis-(2-methyl-5-nitrophenyl) polymerization <tr> <td>(22.) 1,4-bis-(2,4,6-trinitrophenyl)-benzene] polymerization <tr> <td>B. Synthesis reactions with Polyazines <tr> <td>(1.) Hexamethoxybenzene <tr> <td>(2.) 2,4-dinitrophenyl-2,5-dimethyl-3-bromo-benzene] Polyazobenzopyrene <tr> <td>(3.) 2,4,6-trinitrobenzene <tr> <td>(4.) methyl 2 nitro <tr> <td>C. Addition reactions of azophthalates <tr> <td>(1.) 4-nitrobenzoic azophthalate <tr> <td>(2.) 4-(2-nitrophenyl)-benzoic azophthalate <tr> <td>(3.) 4-(2,4-dinitrophenyl)-benzoic azophthalate <tr> <td>(4.) 4-(2,4,6-trinitrophenyl)-benzoic azophthalate <tr> <td>(5.) 4-(2,4,6-trinitrophenyl)-benzoic azophthalate <tr> <td>(6.) 4,4'-(2,4,6-trinitrophenyl)-diazobenzophthalate <tr> <td>D. Synthesis reactions with azophthalates <tr> <td>(1.) 4-nitrophenyl-2,4,6-trinitro-benzene <tr> <td>(2.) 4-nitrophenyl-2,4,6-trinitro-benzene <tr> <td>(3.) 4-nitrophenyl-2,4,6-trinitro-benzene <tr> <td>(4.) Hexamethoxybenzene <tr> <td>(5.) 4-nitrophenyl-2,4,6-trinitro-benzene <tr> <td>(6.) 4,4'-dinitrophenyl-2,4,6-trinitro <tr> <td>(7.) 4,4'-dinitrophenyl-2,4,6-trinitro <tr> <td>E. Synthesis of Di-heterocyclic </td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr></td></tr>	(21.) 1,4-bis-(2-methyl-5-nitrophenyl) polymerization <tr> <td>(22.) 1,4-bis-(2,4,6-trinitrophenyl)-benzene] polymerization <tr> <td>B. Synthesis reactions with Polyazines <tr> <td>(1.) 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E. Synthesis of Di-heterocyclic																							

	Page
(1.1) \mathbb{R} -linears \rightarrow tensor isomorphism	81
(1.2) \mathbb{R} -Bil. Form. - the Lemma	81
(1.3) \mathbb{R} -Bil. Form. - the Lemma	82
(1.4) \mathbb{R} -Bil. Form. - the Lemma	82
(1.5) \mathbb{R} -Bil. Form. - the Lemma	82
(1.6) \mathbb{R}_n -Bil. Form. - the Lemma	82
(1.7) \mathbb{R} -Bil. Form. - the Lemma	82
(1.8) \mathbb{R} -Bil. Form. 1. - the Lemma	82
(1.9) \mathbb{R} -Bil. Form. 1. - the Lemma	82
(1.10) \mathbb{R} -Bil. Form. - the Lemma	82
(1.11) \mathbb{R}_n -Bil. Form. 1. - the Lemma	82
(1.12) \mathbb{R} -Bil. Form. - \mathbb{R} -Bil. Form. 1. - the Lemma	87
(1.13) \mathbb{R} -Bil. Form. - the Lemma	87
(1.14) \mathbb{R} -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.15) \mathbb{R} -Bil. Form. 1. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.16) \mathbb{R} -Bil. Form. 1. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.17) \mathbb{R} -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.18) \mathbb{R} -Bil. Form. 1. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.19) \mathbb{R} -Bil. Form. 1. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.20) \mathbb{R}_n -Bil. Form. 1. - the Lemma	88
(1.21) \mathbb{R} -Bil. Form. 1. - the Lemma	88
(1.22) \mathbb{R}_n -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.23) \mathbb{R}_n -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.24) \mathbb{R} -Bil. Form. - the Lemma	88
(1.25) \mathbb{R}_n -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.26) \mathbb{R}_n -Bil. Form. - \mathbb{R} -Bil. Form. - the Lemma	88
(1.27) \mathbb{R} -Bil. Form. - the Lemma	88
(1.28) \mathbb{R} -Bil. Form. - the Lemma	88

	Page
I. Summary	88
II. context background	99
III. methodology	100
IV. findings	101
V. discussion points	102

APPLICATION OF AFFINITY AND ENTROPY

There are but two possible results from the interaction of two systems: (1.) The two sets are made of molecules may be formed from the two original systems of molecules, or, (2.) The two molecules may combine to form a new, single substance. The latter case requires an analysis of collision reaction, and constitutes the subject of this research.

It is not surprising that such important problems should have resulted in a great number of hypotheses attempting to explain their nature. Although it is impossible to include here a complete discussion of these theories, it becomes pertinent to outline some of the various explanations which have been proposed.

Perhaps the most general view is that promulgated by Michael¹ in his "Molecular Principles". Addition is explained by the result of the affinity of the two interacting systems for each other, and occurs in such a way as to produce the most "stably combined" molecule. This "stabilization of affinity" is assumed to depend principally upon the nature of the atoms comprising the two interacting systems.

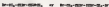
The hypothesis presented by Trautner, et al.² maintains that the criterion which determines that an addition reaction is formed is a difference between the two interacting systems, which he termed a "difference in potential". The greater the difference in potential be-

From them, the more rapid still in the instantaneous sense, the difference in potential may be calculated by assuming the velocity with which the two substances combine. Supposing, there is fixed amount, almost instantaneous, as variable product. This product may undergo a change and be converted into a stable product whose rate of formation is non-variable. It is logical to assume that with the formation of the stable product the affinity is less than in the case of the labile compound.

Wald², in his classical work published in 1899, pointed out the stability of unimolecular systems containing two labile bonds supported by one stable bond. This type of unimolecular system is referred to by the term "conjugated" or "conjugate system", and it is the affinity to conjugate included in this classification distinguishes the system of the present research. In such systems the interaction is limited almost wholly to the two labile atoms of the chain, while the two stable atoms are nearly completely irrelevant. The kinetic theory is based upon the assumption that, in the case of a labile bond between two atoms, the state of the affinity is not much up, but in addition to that which is used in holding the atoms together there is a slight amount which is termed "fractional affinity" or "partial valency". However the labile bonds occur in alternate positions, the combined affinities of the two stable atoms result in a actual interaction. This conception of partial valency is represented by dotted lines, as follows:



Wolcott², in 1953, investigated the addition of bromine to tri-*o*-allyls, which is a typical conjugated system. Since this conjugated system is alternately pair of double bonds, it is to be expected that it could react with two atoms of bromine simultaneously, or at least take up the atoms of bromine at one of the double bonds, as follows:



Wolcott reported that there was first formed a liquid dibromide which soon changed into a solid, melting point 59-60° C. Wolcott², proved that this solid is a 1,4-dibromo-1,3-butadiene-2, which contains a double bond in the center of the chain,



Wolcott and Jones³, showed that the addition of three bromine to a conjugated triene, after open treatment of the dibromide with potassium hydroxide, there is generated a 1,3-diene which in which the double bonds are again located in the 1,3-position. Subsequently, Jones, Lawrence and Wolcott⁴, have confirmed Wolcott's report that a liquid dibromide is first formed. They have shown that this is a 1,4-dibromo-1,3-butadiene-2, which changes reversibly at 100° C. into a 1,3-dibromo-1,3-butadiene-2, and remains in an equilibrium mixture containing 80% of the former and 20% of the latter.

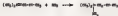
The present research deals more specifically with reactions in cyclic, heterocyclic systems. Wolcott has shown that in such cases, where the different types of double bonds are conjugated with each other,



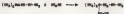
addition agents such as hydroperoxides and hydroperoxide groups will add to the ends of the conjugated diene, a subsequent transformation from the end to the other side may occur:



Thus in this reaction two different types of macroradicals in a single molecule is formed. It is most interesting to observe the reaction of agents which are capable of adding twice both the carboxyl group and the styrene side linkage. Acrylic acid, for example, reacts with conjugated diene, a typical alpha, beta-unsaturated ketone, it affords only the styrene linkage, leaving the carboxyl group intact:



Hydroperoxides, on the other hand, produce two different products according to the conditions. Burton, et al.^{10,11,12} observed that when the reaction is carried out in solution solution in the presence of various solvents, the principal product results from the 1,4-addition of hydroperoxides, with subsequent rearrangement:



However, if hydroperoxides hydroperoxide in anhydrous dichloromethane with various solvents and then reacted with conjugated diene

where α is the angle of rotation, the angular velocity ω is constant and equal to $\dot{\alpha}$.

$$(\mathbf{M}_0)_y = (M_0 \sin \alpha - \dot{\alpha} t) - \omega t$$

It is apparent that in natural rotation the angular velocity is the same constant, but in arbitrary rotation the angular velocity becomes arbitrary and arbitrary to it are substituted.

One of the main characteristics of conjugate systems is the transformation of natural rotation from one axis to another. In the preceding part it has been shown that when a rigid body is rotated about a fixed axis which is the natural axis of the conjugate group, the rotation is the same about the other axis of the conjugate group; the fact that one axis is a constituent of the conjugate group means it is in a position of an natural rotation. Similarly, the rotation about a fixed axis instead of the upper axis of the conjugate group. It is a well known that the rotation is arbitrary, and the addition of rotation to rotation, this place in the L_2 -position with subsequent reorientation in the L_2 -position, or the other way. However, it is important to note that there is an alteration of velocity, and that alternate axes apparently require a similar velocity and a similar position.

In 1924,¹² in 1925, proposed that in such circumstances the inertia group is transferred from the axis of rotation to the axis of rotation, and that the inertia group might move along a state of arbitrary rotation and double-axis rotation, the angular and angular velocity changing places in the path of the inertia group. This hypothesis,

which substituted analogous properties to only the alternants which in the chain, opened up a new perspective of conjugation and furnished a foundation for all subsequent investigations in this field.

The systems which Hückel utilized in interpreting his theory of partial valences are not in accordance with and cannot be converted directly into the systems which are used in the modern electronic interpretation of valence. Thus, as is shown in 14 , 15 (see also a comprehensive account of work in the calculation of these orbitals, but the complete electronic interpretation of conjugated systems is first proposed by Long¹⁶ in 1934, in addition with the theory of polar activation of aromatic bonds. It is proposed that the only orbitals which undergo structural change are the orbitals that, and their addition reactions of electrons under the influence of polar reagents are dependent upon the localization of one of the bonds of the double bonds:



It is a characteristic property of conjugate systems to develop a series of positive and negative charges on alternants when as a result of the activation by the double bonds:



In the former form of the molecule there is a complete alternation of single and double bonds, but in the activated form there are converted into a series of atoms bearing alternately positive and negative charges. This interpretation, in which local charges of like sign are developed by

alternate sites, is entirely in accordance with the hypothesis of hyperconjugation.

In many cases, addition to unsaturated compounds have been attributed to a polar contraction of the double bond²⁷, this is brought about under the influence of an ionic reagent, and gives rise to an unstable non-polar bond:



The arrows indicate that, as a result of the presence of substituents, a pair of shared electrons becomes transferred from the double bond to the site of substituents which acquire a net negative charge in the contracted product. In the unsaturated aldehydes and ketones the sign of the charge is determined by the tendency of the oxygen to acquire a negative charge, and not the carbon atoms, because the oxygen atom at the end of the double bond acquires, and the remaining carbon atom acquires positive and negative:



A more detailed discussion of the electronic interconversion of conjugated systems must be presented within the limits of this discussion; however, the material herein presented is sufficient to indicate that conjugated systems possess the property of undergoing a rearrangement of bonds in such a way that a delocalized electron is transferred progressively

from one set of the double to the other. This provides for the interconversion of the symmetric character of an isolated fragment as both the $1,2$ - and $1,4$ -additions, and provides a mechanism for the transformation of a $1,2$ -addition into the symmetric $1,4$ -addition.

Blaise's original classification of conjugate systems included only those exhibiting allylic and vinylic groups. Johnson¹⁸ and Jander¹⁹, have shown that any element, for example nitrogen and sulfur, which is capable of forming a double covalent bond, or which possesses unshared electrons, may form an integral part of a conjugate system. Johnson¹⁸, succeeded in classifying such systems into sharply defined groups, since their reactions are largely dependent upon the electrical nature of the sites which are present. His classification is as follows: (1.) poly-valent systems, (2.) hetero-valent systems, (3.) hetero-valent systems, (4.) univalent systems, (5.) disubstituted polyene systems. In the field of this classification, allylic, hetero-substituted hetero, vinyl are the subject of this research, being in the hetero-valent system. These systems include hetero-atom vinyl bond in substituted alkenes, and in an allylic position carbon halides. Development of vinylic character occurs in the following manner:



Involvement of the vinylic electron in these systems is conditioned by the fact that the hetero-atom (oxygen) forms a more stable anion than the carbon atom.

The effect of substituent groups upon addition to double bonds

was investigated by Iser²¹ in 1970, he concluded that it is possible to afford addition reactions in this manner. Later, Jones²² undertook similar investigations and succeeded in identifying Iser's conclusions. In the course that of methyl, methoxy, or propyl groups, or bromine atoms are connected to the vicinity of a double bond, bromine does not readily add. For example, bromine does not add to methacrylylic acid,



nor to diene-acrylate acid,



although both contain double bonds.

The investigations of Iser, et al.^{21,22}, also showed that other cyclic, methoxy, and propyl groups are placed in the vicinity of a double bond, the effect upon the addition of bromine is in the order: cyclic > methoxy > propyl. The influence of the propyl group is also diminished by substitution on it; it is weakened by chlorine, although a nitro group in the ortho-position has the least effect, and one in the para-position the most effect. Furthermore, he was able to show that the addition of bromine to double bonds is reversible, and that the equilibrium shift is obtained in various stages in dependent upon the substituents in the molecule. Interestingly he has also shown that there is a remarkable correlation between the effect of substituents on the addition of bromine and their influence upon the dissociation constant of acids.

a review of the literature reveals a scattered host of investigations concerning the influence of substituents upon the addition to conjugate systems, and especially alkenes, heteroconjugated systems. It is with the object of clarifying this problem that the present research has been undertaken. The present theories and experimental approaches are quite insufficient, and a single field exists for theories.

Tucker and Wills^{1,2} found that benzocyclohexene reacts with aliphatic amines in the cold to give the compound,



In a similar manner addition compounds of benzocyclohexene with several primary aromatic amines were prepared. They were not successful in adding secondary amines as direct secondary bases. The addition goes readily in the cold, generally with or without a catalyst. Generally the addition does not occur in one step. The series of benzocyclohexene and one mole of amine, one mole of nitrobenzidine ($\text{C}_6\text{H}_5\text{N}_2\text{C}_6\text{H}_5 \approx 4\text{m}^2$), one mole of 1,4-benzidine, or one mole of alpha-naphthylamine, however, epinephrine quantities of the ketone combine with maline, p-toluidine, and beta-naphthylamine. These compounds are pure white; however, the introduction of a chromophore group (e.g., nitro group) into the amine results in a color change to red or yellow.

It is noteworthy that Tucker and Wills added p-toluidine to benzocyclohexene, but were unsuccessful in adding o-toluidine; appar-

only the position of methyl groups on the benzene rings does affect the solution. Although some compounds were found to be stable under ordinary conditions, it is stated that by attempting to synthesize benzoin-oxaphenone oxides, or by short heating, total decomposition occurred.

Georgi and Göttsche²² attempted to add hydrochloric acid to benzoin-oxaphenone by mixing in alcohol with sodium hydroxide. The expected product did not occur and instead, dihydroxyacetophenone, $\text{HO(CH}_2\text{OH)COCH}_3$, melting point 87° , was formed. This dihydroxy ketone was used, $\text{C}_8\text{H}_{10}\text{O}_2$, dihydroxyacetophenone.



was obtained in the form of prismatic red crystals, melting point 10° & then heating with sulfur it decomposed into the original substances; however, the hydrochloride, melting point $115-116^\circ$ &, was prepared. The same and dihydroxyacetophenone in the presence of alkali is not constant, and polyhydroxyacetophenone is the product to be isolated.

NOMENCLATURE

Several systems of nomenclature for pyranosides and disaccharides exist in the literature. However, it seems expedient to utilize throughout this discussion the system adopted by Chemical Abstracts.

The official Chemical Abstracts formula for pyranosides is:



Directions in which the substituent groups are attached to the carbon atoms are termed 1,2-distributions or 1,3-distributions of pyranosides. The former system is used throughout the course of this discussion.

The formula given by Chemical Abstracts for disaccharides is:



The positions of substituents on the pyranose rings are indicated by the proper numbers. Substituents on the aliphatic carbon atoms are indicated by the Greek letters alpha and beta. In the case of pyranose disaccharides are named according to the anomeric substituents and between them which they are prepared, using either numbers or the Greek letters, alpha and beta to indicate the positions of substituents on the pyranose rings. For example,



by the formal names *p*-chlorobenzophenone or 4-chloro-2,2'-(1-oxo-2-phenyl-2-phenyl)propane. Because of its lack of toxicity and toxicity data which is not used in the present screening format, it is frequently mentioned in the literature.

The compounds which have been prepared in this research are used in derivatives of pyrethrin, providing the name of the chlorine or its equivalent in its addition compound. For example,



is formal 1,4-dia-(1-oxo-2-(4-chlorophenyl)propanoate) pyrethrin.

The official chemical abstracts describe the synthesis as:



The addition compounds of pyrethrin are used in derivatives of pyrethrin, providing the name of the chlorine or its equivalent in the

one of the pyruvate addition reactions. For example,



is designated as [1-oxoethyl] addition.

THEORETICAL SIGNIFICANCE

The object of this investigation, as outlined in the foregoing paper, is to study the effect of substituted groups upon addition to alpha, beta-unsaturated ketones. Many unsaturated ketones of the acyclic series offer a greater variety of substitutable than those of the cyclic series, the investigation is limited chiefly to this group. In order to make a comparison of results, only two aliphatic groups were used throughout the course of the research. The heterocyclic secondary amines, piperidine, and the heterocyclic secondary amine ether, morpholine, were chosen for two reasons; (1.) addition of these compounds are reported in the literature; and (2.) the compounds which should react most readily with addition would be similar in structure to certain apparatus and apparatus, and should prove to be interesting in their physiological action.

THEIR for simplicity, unsaturated ketones were to be prepared, to illustrate a tendency to prepare most of the intermediates used in the investigation. Most work of this kind in the literature is extensive and will not be reproduced, a detailed account of the properties of these compounds is presented in the addition article, description of intermediates.

Unsaturated ketones of the acyclic series are easily readily prepared by the classical olefin reaction. Anti-selective preparation of the corresponding acyclic aldehydes and acyclic ketones were obtained in a suitable quantity of the alcohol. The isolated gram of a 10% aqueous solution of sodium hydroxide per mole of aldehyde (or ketone) was then

which the solution was thoroughly stirred, and the alpha, beta-unsaturated ketone between generally separated out almost immediately. In most cases it was in the form of a pale yellow solid. If the cultured yeast reaction, air-dried, and then recrystallized from commercial hexane or 70% ethanol, depending upon the specific nature of the compound. Each of the alpha, beta-unsaturated ketones used in this investigation are chiral, and their preparation may be indicated by the following type reactions:



where R^1 and R^2 represent any alkyl (or substituted benzene) groups.

The addition of pigments to the alpha, beta-unsaturated ketones was prepared and found to be best effected by refluxing the reaction in toluene solution. Contrary to the findings of Gordon, et al., *M., 12, 44*, which did not appear to substantiate their method. In general, the procedure was to dissolve one mole of substrate pigments in toluene (where necessary) as soon as solution was complete, a toluene solution of the ester of the alpha, beta-unsaturated ketone was added. The solution was then refluxed over a Dean-Stark for ten hours, although in each case a shorter reflux-time would have proved sufficient, this period of time was deemed safe for all additions and was used throughout for the sake of consistency. The solution was then allowed to stand in the ice box for at least one day in some cases, where the product was still in suspension, it was allowed to stand longer. Generally, the addition compound separated out in the form of crystals, which on filtration

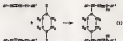
crystals. These were filtered off from the solution with suction, washed with a small amount of HCl ethanol to remove the telomers, and then washed with a large amount of water in order to remove any unreacted glycerol. The compound was then dried.

The yields of the crude products varied from 70% to 90%, but in each case approached the better value. Telomers are found to be both useful as a recrystallizing solvent, generally it was necessary to recrystallize the addition compound at least three times before subsequent recrystallization gave an increase in melting point. The pure product in each case was in the form of crystals, pure white crystals. A considerable loss of product was incurred in the recrystallizing process, and the yields of the purified products dropped to the at one-third of the per cent yield of the crude products. However, a more suitable solvent could not be found, and it was desirable to utilize only the highly purified products for analysis.

Other and nitrogen determination were carried out by the combustion method in the case of the parent compound, 1,4-bis-phenylene glycerol, in order to definitely establish its composition. The nitrogen content was determined also, by means of the Kjeldahl method. Analytical data for the other addition compounds were sufficient to establish their composition, since these were considered sufficient to characterize the individual compounds.

addition of glycerol to diisocyanate probably occurs in the usual manner of addition to alpha, beta-unsaturated ketones. Glycerol is able to the two end partial reactions (1,4-position) to give the

molecule from (I) of the addition compound, where α' and α'' are any groups:



The stable form, being unstable, is rapidly converted to the more stable (11), with which is then subject to reversible epimerization:



When epimerization is finished, the major portion of the compound exists in the stable beta form (11), and the resultant product is effectively a 1,4-addition compound, although a non-additive compound,



and various stereoisomers are theoretically possible besides the isomeric form present, only the latter compound was isolated in each case.

The same isomer addition has been effected and isolated in the experimental series; a great variety of substituted alcohols have undergone addition-elimination reactions, alcohols bearing methyl, ethyl, allyl, vinyl, ethoxy and other groups in either one both because rings have undergone addition. The position of the substituted group on the ring apparently had no demonstrative effect.

The experimental conditions, "negative reactions with pyridine", indicated the alpha, beta-unsaturated ketones which did not yield addition compounds under the experimental conditions which were successful with the foregoing compounds.

From the nature of these compounds it is to be expected that they display a limited solubility in most solvents. They are insoluble in water, alcohol, ether, and hydrocarbons and slightly soluble in cold benzene and toluene, and fairly soluble in the hot solvents. They are moderately soluble in halogenated solvents such as chloroform, dichlorobenzene and carbon tetrachloride.

The compounds are all stable under ordinary conditions, and are not decomposed by heating with water or in the case with the pyridine addition compound reported by Deerp and Volmer²⁰. The 1,4-dimethyl-2-penten-3-one was heated with hydrochloric acid in one completely decomposed into pyruvic acid, acetaldehyde and alcohols. A similar decomposition occurred also for hydrogen chloride was passed into a chloroform solution or hot toluene solution of the compound; hence, a hydrochloride of the compound was not isolated. Thus a toluene solution of

1,4-dia-oxalones prepared via refined vinyl vinyl acetate, decomposi-
 tion again resulted instead of the expected formation of the quaternary
 ammonium base,



a small amount of a pale brown compound separated from the left solution.
 This was filtered off and recrystallized from a-hydroxyethyl acetate
 yields a solid 15 to be identical with the product obtained by refluxing
 vinyl acetate with pyridine in toluene solution. The 15,16-bis are sep-
 arated to form a 2:1 ratio, leaving a pale yellow solid, which
 was recrystallized from the solvent. Mixed melting points proved it to
 be the unreacted ketone, thalidomide. These decompositions are perhaps
 surprising, since the unreacted ketone remains however, further re-
 flexion reveals that this is entirely analogous to the usual degradation
 of ketone, whereby the vinyl group is split off as an unreacted ethyl-
 ether.

The addition of a crystalline to the vinyl, beta-unsaturated ac-
 etate was found to be best effected by refluxing an excess of acrylonitrile
 with the ketone in commercial toluene. In general, the procedure was to
 dissolve two-thirds mole of the ketone in a stoichiometric amount of boiling
 commercial toluene under reflux an excess of acrylonitrile was complete three-
 fourths mole (two-thirds mole excess) of acrylonitrile crystalline was added.

The reaction was then continued over a three hour for two hours, as was done in the addition of pyridine. The reaction was allowed to stand in the ice box over the night, whereupon the addition compound separated out in the form of pure white, needle-like crystals. These were filtered off from the liquid with suction, washed with a small amount of dry ethanol to remove the liquid, and then washed with a large amount of ether in order to remove any unreacted acrylonitrile. The compound was then dried.

The yields of the crude products were very nearly quantitative, all being well over 90%. Crude liquid butadiene was found to be best suited as a recrystallizing solvent generally if it was necessary to recrystallize the addition compound at least three times before attempted recrystallizations gave an increase in melting point. The pure product in all cases was in the form of pure white, needle-like crystals. An appreciable loss of product occurred in the recrystallizing process, but it was considered advisable to collect the compounds in separate portions before recrystallizing them.

Analytical data for the acrylonitrile addition compound was compared to average determinations by the Kjeldahl method, since the nature of the addition was satisfactorily proved in the addition of pyridine.

The mode of addition of acrylonitrile to the alpha, beta-unsaturated ketone is relatively quite analogous to the addition of pyridine, involving a 1,4-addition with a subsequent anti-bond rearrangement to give the 1,2-addition product. However, in the case of the acrylonitrile addition, the mode of acrylonitrile and the mode of the structural ac-

are involved. The cases in which addition has been effected with acrylonitrile are indicated in the experimental tables. Glycerol bearing methyl, ethoxy, nitro and brom groups as often as both benzene rings have undergone addition. Here again, the position of the substituent group on the ring apparently has no determinative effect.

The experimental results obtained, therefore combined with synthesis, indicate the alpha, beta-unsaturated ketones which did not yield addition compounds under the experimental conditions which were successful with the foregoing compounds. In most cases the addition compound apparently was formed, but was so unstable that attempts to purify it resulted in complete decomposition to acrylonitrile and the corresponding ketone.

Stability properties of the acrylonitrile addition compounds are quite similar to those of the pyridazine compounds. They are thick, white to color, but soluble in alcohol, ketones, chloroform and acetone, and very soluble in dilute hydrochloric acid. They are slightly soluble in cold concentrated sulphuric, and readily soluble in the hot extract.

Under ordinary conditions the compounds which were isolated are quite stable. However, like the pyridazine compound reported by Hantzsch and Schmitt¹¹, and unlike the pyridazine addition compounds, they are decomposed by heating with water; the decomposition results in acrylonitrile and the unsaturated ketone. Treatment with hydrochloric acid results in a similar decomposition.

EXPERIMENTAL TABLE AND DISCUSSION

4. Physical constants of piperazine

4. Preparation of 1,4-dia-*trans*-piperazine

Diethylene and tetraethylene glycol (2.0 mole) of anhydrous piperazine was dissolved in a solution consist of boiling toluene under reflux. A hot toluene solution of 20.0 gram (0.4 mole) of diethylene (trans-*trans*-isomerism) was then added. The total volume of the solution was about 200 ml. The solution was refluxed over a few hours for ten hours and then allowed to stand in the lab for one day, changed a syringe, which precipitate resulted. The suspension filtered at 60°C., washed with a small amount of dry ethanol and then with a large amount of water. It was stirred and recrystallized from toluene three times, yielding white, pure white crystals. These were washed with ether after the final recrystallization, in order to remove the toluene. The yield of crude product was 27.0 gram (70% of theoretical) and the yield after three recrystallizations was 20.0 gram (50% of theoretical). The crude product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. This material for nitrogen is given 0.400 g. calculated: 0.370 g. carbon analysis gave 0.100 g. calculated: 0.100 g. The hydrogen analysis gave 0.100 g. calculated: 0.100 g. The corrected melting point of the pure compound was 124.0-124.5° C.

b. 1,4-dic-*trans*-polymer.

Optical formula

$\frac{1}{2}[\alpha]_D^{20}$

Structural formula



Molecular weight

200.27

rotating power

$10000-200.27^2 \alpha$

Yield of crude product

50g

Yield after three recrystallizations

50g

analysis

found calculated

Carbon

61.0% 61.0%

Oxygen

31.0% 31.0%

Hydrogen

8.0% 8.0%

solubility

Water

insoluble

Alcohol

insoluble

Ether

insoluble

Benzene

slightly soluble

Toluene (recrystallizing solvent)

slightly soluble

Chloroform

very soluble

Carbon tetrachloride

soluble

5. Preparation of 2,4-dipic(2-hydroxyethoxy) pyrazine-

Four and three-tenths grams (0.02 mole) of adipic acid was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 25.0 grams (0.1 mole) of 2-hydroxyethoxy was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the ice box one day, whereupon the pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 50% ethanol and then with a large amount of water. It was air-dried and then recrystallized once from about 10 liters of toluene. This yielded a pale yellow, crystalline compound, which became light brown upon standing in a desiccator. The crystals were washed with ether after the final recrystallization in order to remove the toluene. The yield of crude product was 24.7 grams (87% of theoretical) and the yield after one recrystallization was 1.0 gram (3% of theoretical). The purified product was dried in vacuum over some anhydrous calcium chloride and before being subjected to analysis. The corrected for nitrogen it gave 0.480 g. calculated, 0.480 g. the corrected melting point of the pure compound was 145.0-146.0° C.



6. 1,4-bis(2-hydroxyethyl) piperazine

Molecular Formula



Structural Formula



molecular weight

188.27

boiling point

188.0-188.0° C.

heat of vaporization

57g

heat of fusion for polymerization

57g

analysis

found	calculated
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C, %

61.40	61.40
-------	-------

solubility

water

insoluble

alcohol

insoluble

ether

insoluble

benzene

slightly soluble

nitrobenzene (polymerizing solvent)

slightly soluble

dichloroethane

fairly soluble

dimethylacetone

fairly soluble

6. Preparation of 1,4-dioxane-2-thione-thiazole-5-pyrimidinone.

Four and three-halves grams (0.05 mole) of sulphurous pyrimidinone was dissolved in a minimum amount of boiling toluene under reflux. A hot filtered solution of 24.8 grams (0.1 mole) of 2-thiopyridone was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the ice box for three days, whereupon a white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was air-dried and then recrystallized three times by dissolving in a minimum amount of boiling toluene and then adding a large amount of heptane. The compound separated out immediately. It is insoluble in concentrated hydrochloric acid, whereas 2-thiopyridone, which is the principal impurity, is quite soluble. The compound was washed with ether after the final recrystallization. This yielded about pure white crystals, which became brownish yellow upon standing in a desiccator. The yield of crude product was 24.8 grams (50% of theoretical) and the yield after three recrystallizations was 8.1 grams (20% of theoretical). The product melted and dried in vacuo over concentrated sulphuric acid before being subjected to analysis. The compound for nitrogen is 4.96% 4.96% g. calculatedly 4.96% g. The corrected melting point of the pure compound was 138.5-139g^o g.

h. 1,4-Bis-(2-methoxy-phenyl) piperazine

Empirical formula

$C_{16}H_{20}N_2O_2$

Structural formula



Molecular weight

272.37

Melting Point

100-101°C^o b.

Color of solid product

off

Color after three recrystallizations

off

Analysis

found calculated

C, %

64.66 64.66

H, %

N, %

insoluble

O, %

insoluble

solub

insoluble

water

partly soluble

alcohol

partly soluble

acetone (recrystallizing solvent)

insoluble

chloroform

soluble

dichloromethane

soluble

4. Preparation of 1,4-diox-4'-chlorobenzene) pyrene.

Four and three-halves grams (9.00 mole) of sulphuric pyrene was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 20.1 grams (9.1 mole) of 4'-chlorobenzene was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for two hours and then allowed to stand in the ice box two days, whereupon a white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of dry ethanol and then with a large amount of ether. It was air-dried and then recrystallized three times by dissolving in boiling toluene and then cooling in the ice box. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization, in order to remove the toluene. The yield of crude product was 20.8 grams (77% of theoretical) and the yield after three recrystallizations was 14.8 grams (57% of theoretical). The purified product was dried in vacuum over concentrated sulphuric acid before being subjected to analysis. When analyzed for chlorine it gave 6.46% Cl. Calculated: 6.47% Cl. The corrected melting point of the pure compound was 129.5-130.5° C.

4. 1,4-bis-(4'-oxydiphenylacetone) glycerol.

Empirical formula

$C_{20}H_{20}O_4$

Structural formula



Molecular weight

324.37

Melting point

127.5-128.5°

Yield of crude product

70%

Yield after three recrystallizations

47%

Analysis

Found calculated

C, 61.73%

61.60% 61.60%

Properties

Water

Insoluble

Alcohol

Insoluble

Ether

Insoluble

Acetone

Partly soluble

Chloroform (recrystallizing solvent)

Partly soluble

(4) Insoluble

Insoluble

(5) Insoluble

Insoluble

B. Preparation of 2,4-bis-(3-methyl-2-butenyl) diglycidyl ether.

Four and three-tenths grams (0.01 mole) of anhydrous aluminum chloride was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 44.3 grams (0.3 mole) of bischloroacetalone was then added. The total volume of the solution was about 150 ml. The solution was refluxed over a fire flame for ten hours and then allowed to stand in the lab for three days, changing a pair of filter, separating compound separated out. The compound was filtered with suction, washed with a small amount of 50% ethanol and then with a large amount of water. It was air-dried and then recrystallized three times from toluene. This yielded a pure white, flaky compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 11.3 grams (70% of theoretical) and the yield after three recrystallizations was 8.3 grams (40% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. When analyzed for chlorine it gave 6.00% Cl. Calculated: 6.00% Cl. The corrected melting point of the pure compound was 140.0-147.0° C.

4. 1,4-Dioxane(2-chloroethanol) polymer:

Empirical formula



Structural formula



Molecular weight

174.17

Melting point

120-120.5° C.

Heat of fusion (cal/g)

7.0

Heat of fusion (kcal/mole)

1215

Analysis

Found: calculated

C, %

6.85 6.85

Calculation

Water

Insoluble

Alcohol

Insoluble

Ether

Insoluble

Diester

Partly soluble

Diester (saponifying solvent)

Partly soluble

Diester

Partly soluble

Diester

Partly soluble

5. Preparation of 1,4-dia-(3,4'-dichloro-benzene) piperazine

Four and three-halves grams (0.02 mole) of adipic acid and 2.46 grams (0.02 mole) of 1,4-dia-(3,4'-dichloro-benzene) piperazine were dissolved in a solution consist of boiling toluene under reflux. A hot toluene solution of 25.7 grams (0.1 mole) of 1,4'-dichloro-benzene was then added. The total volume of the solution was about 170 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the lab for three days, changing a pale yellow, crystalline material separated out. The material was filtered with caution, washed with a small amount of 95% ethanol and then with a large amount of water. It was dried and then recrystallized three times from toluene. This yielded a pure white, crystalline material. The crystals were washed with ether after the final recrystallization. The yield of crude product was 21.0 grams (82% of theoretical) and the yield after three recrystallizations was 8.1 grams (30% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. This material had nitrogen 11.66% (calculated, 11.88%) C, 51.66% (calculated, 52.4-52.5%) H. The corrected melting point of the pure material was 128.4-128.5° C.

6. 1,4-bis-(2,4'-diaminodiphenyl) glycolate.

Empirical formula



Structural formula



Molecular weight

320.40

Melting point

226-228°C. 5.

Yield of crude product

4g

Yield after three recrystallizations

3g

Solubility

poor soluble

in water

4.5% 4.5%

miscibility

water

variable

ethanol

variable

ether

variable

benzene

slightly soluble

nitrobenzene (recrystallizing solvent)

poorly soluble

chloroform

slightly soluble

dichloroacetone

poorly soluble

7. Preparation of 4,4'-dian(4'-bromo-bisoxazole) pyromazine.

Four and three-tenths gram (0.10 mole) of anhydrous pyromazine was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 18.7 gram (0.1 mole) of 4'-bromo-bisoxazole was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a 10-cm. flask for ten hours and then allowed to stand in the ice box for four days. A pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with ethanol (95%) and then with a large amount of water. It was dried and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 16.8 gram (70% of theoretical) and the yield after three recrystallizations was 15.7 gram (68% of theoretical). The purified product was dried in vacuo over anhydrous calcium chloride before being subjected to analysis. This analysis for nitrogen (I gave 4.48% N, calculated: 4.46% N. The corrected melting point of the pure compound was 114.8-117.5° C.

1. 1,4-gua-(*o*)-guac-acetone) guaymasan.

Empirical formula

$C_{10}H_{16}O_4$

Structural formula



Molecular weight

200.0

Melting point

100.5-101.5° C.

Yield of crude product

70g

Yield after three recrystallizations

30g

Analysis

Found calculated

C, 60.0%

60.0% 60.0%

solubilities

water

insoluble

alcohol

insoluble

ether

insoluble

benzene

slightly soluble

chloroform (strongly fluorescing extract)

slightly soluble

diethyl ether

soluble

dioxane

soluble

5. Preparation of 1,4-bis-(1-hydroxyethylamino) piperazine.

One and three-tenth grams (0.01 mole) of cuprous pyruvate was dissolved in a suitable amount of boiling toluene under reflux. A hot toluene solution of 20.0 grams (0.1 mole) of 1-hydroxyethylamine was then added. The total volume of the solution was about 100 ml. The reaction was followed over a few days for two hours and then allowed to stand in the ice box one day, whereupon a white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was air-dried and recrystallized four times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of white product was 10.0 grams (50% of theoretical) and the yield after four recrystallizations was 8.0 grams (40% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 4.00% N. Calculated, 4.44%. The corrected melting point of the pure compound was 101.5-102.0° C.

8. 1,4-bis-(4-oxocyclohexyl) ethane.

Synonym: Nemis

C₁₂H₂₀O₂

Structural formula



molecular weight

200.30

boiling point

168.5-169.0° C.

yield of crude product

50%

yield after pure recrystallization

50%

soluble

water insoluble

in benzene

soluble

miscibility

water

insoluble

alcohol

insoluble

ether

insoluble

acetone

fairly soluble

nitrobenzene (recrystallizing solvent)

fairly soluble

chloroform

soluble

dichloroethane

soluble

9. Preparation of 1,4-diacetyl-4'-acetyl-stilbene

Five and three-fourths grams (0.05 mole) of sodium piperazine was dissolved in a solution amount of boiling toluene after reflux. A hot toluene solution of 25.1 grams (0.1 mole) of 4'-acetyl-stilbene was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the ice box for days, through the alcohol gave white, acrylonitrile compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was separated and then recrystallized three times from toluene. This yielded a pure white, acrylonitrile compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 25.0 grams (80% of theoretical) and the yield after three recrystallizations was 12.1 grams (40% of theoretical). The purified product was dried in water over concentrated sulfuric acid before being subjected to analysis. The analysis for nitrogen at 25 gave 8.20% (calculated, 8.20%) N. The corrected melting point of the pure compound was 121.5-122.5° C.

4. 1,4-Bis-(4-hydroxyphenyl) glyoxal

Empirical formula



Structural formula



molecular weight

230.23

melting point

150.5-151.0° C.

Yield of crude product

40%

Yield after three recrystallizations

4%

analysis

found: calculated

C, %

64.7% 64.7%

nitrogen

water

insoluble

phosphorus

insoluble

chlorine

insoluble

potassium

partly soluble

nitrogen (microanalytical method)

partly soluble

nitrogen

insoluble

nitrogen

partly soluble

10. Preparation of 1,4-bis-(1-methyl-ethyl) piperazine.

One and three-tenths grams (3.60 mole) of nitrogen piperazine was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 24.0 gram (0.2 mole) of 1-methyl-ethylamine was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the ice box one day, changing a white, crystalline compound separated out. The compound was filtered with caution, washed with a small amount of 95% ethanol and then with a large amount of water. It was dried and then recrystallized four times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the third recrystallization. The yield of crude product was 24.0 gram (70% of theoretical) and the yield after four recrystallizations was 4.0 gram (20% of theoretical). The purified product was dried in vacuo over anhydrous calcium chloride before being subjected to analysis. The analysis for nitrogen is given in Table I. The melting point of the pure compound was $107.8-107.9^{\circ}$ C.

8. 1,4-Bis-(2-methoxy-phenyl) glyoxal.

Empirical formula

$C_{16}H_{20}O_4$

Structural formula



Molecular weight

300.36

Boiling point

220.5-223.1° C.

Yield of crude product

70g

Yield after four recrystallizations

30

Analysis

Found Calculated

Carbon

6.50% 6.40%

Microanalysis

Water

Insoluble

Alcohol

Insoluble

Ether

Insoluble

Benzene

Partly soluble

Toluene (recrystallizing solvent)

Partly soluble

Chloroform

Very soluble

Dichloroacetone

Soluble

14. Preparation of bis-(bis-(2,4-dichloro-6-tert-butyl) pyrimidin-

one and thioaromatic group (2,4,6) units) of nitrogen pyrimidinone derivatives by a mixture method of boiling toluene under reflux. A hot toluene solution of 88.5 gram (0.5 mole) of 2,4-dichloro-6-tert-butyl one was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the ice box for two days, through a white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 10% ethanol and then with a large amount of water. It was dried and then recrystallized once from about 150 ml. of toluene. This yielded a pure white, fluffy, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 88.5 gram (100% of theoretical) and the yield after the recrystallization was 44.3 gram (50% of theoretical). The purified product was dried in water over concentrated sulfuric acid before being subjected to analysis. This analysis for nitrogen is given Table I. Molecular weight 282. The corrected melting point of the pure compound was 173.5-175.5° C.

22. 1,4-bis-(2,6-di-*n*-butyl-3-pyridyl) pyrene.

Empirical formula



Structural formula



molecular weight

504.62

melting point

170.4-170.6° C.

yield of crude product

10%

yield after the Borey collection

5%

analysis

found: calculated

nitrogen

4.20% 4.20%

solubilities

water

insoluble

alcohol

insoluble

ether

insoluble

benzene

slightly soluble

toluene (concentrated solvent)

slightly soluble

chloroform

slightly soluble

carbon tetrachloride

fairly soluble

14. Synthesis of 1,4-bis-(2-methoxy-4'-methoxyphenyl) piperazine.

Over and three-hundred gram (0.18 mole) of adipic acid was dissolved in a volume equal of boiling toluene under reflux. A hot toluene solution of 44.8 gram (0.1 mole) of 2-methoxy-4'-methoxyphenol was then added. The total volume of the solution was about 100 ml. The mixture was refluxed over a five days for ten hours and then allowed to stand in the jar for ten days, whereupon a white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was dried and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 44.8 gram (75% of theoretical) and the yield after three recrystallizations was 4.8 gram (8% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 4.76% N. Calculated, 4.75% N. The corrected melting point of the pure compound was 129.5-130.5°

6.

16. 1,4-(1,4-oxadiazole-2,5-diyl-2-oxoethylidene) polymerization.

Empirical formula



Structural formula



Molecular weight

260.28

Melting point

207.5-208.5° C.

Yield of crude product

70%

Yield after three recrystallizations

47%

analysis

found calculated

nitrogen

6.76% 6.76%

solubility

water

insoluble

alcohol

insoluble

ether

insoluble

benzene

partly soluble

toluene (recrystallizing solvent)

partly soluble

chloroform

soluble

dichlorobenzene

soluble

III. Preparation of 1,4-Bis-(4-carboxy-6-bromo-styrene) polymer

Four and three-hundred grams (1.66 mole) of styrene polymer was dissolved in a solution amount of boiling toluene water reflux. 1.66 grams solution of KLF gram (1.66 mole) of 4-carboxy-6-bromo-styrene was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a three hours and then allowed to stand in the ice box and top, through a glass funnel, crystalline compound separated out. The compound was filtered with vacuum, washed with a small amount of 95% ethanol and then with a large amount of water. It was air-dried and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 10.6 grams (64% of theoretical) and the yield after three recrystallizations was 3.5 grams (22% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 5.88% N, calculated: 5.89% N. The corrected melting point of the pure compound was 188.0-188.5° C.

25. 1,4-bis[2-(methoxyethoxyethoxyethoxy)] piperazine.

Empirical formula



Structural formula



Molecular weight

404.52

Refining point

150.0-155.0° C.

Yield of crude product

60%

Yield after three recrystallizations

7%

Analysis

Found Calculated

Carbon

57.64% 57.64%

Microanalysis

Water

Insoluble

Miscel

Insoluble

Alcoh

Insoluble

Alkane

Partly soluble

Alkane (methylcyclohexane)

Partly soluble

Dioxane

Partly soluble

Dichloroac

Partly soluble

18. Preparation of 1,4-bis(2-methyl-2'-bromo-ethoxy) pyrene.

Four and three-tenths gram (0.02 mole) of sodium pyrene was dissolved in a solution normal of boiling toluene under reflux. A hot toluene solution of 20.1 gram (0.1 mole) of 2-methyl-2'-bromo-ethoxy was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a three finger for ten hours and then allowed to stand in the jar for two days, sometimes a pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was air-dried and then recrystallized five times from toluene, each yellow crystals of the separated substance contained 16 separate molecules with the fifth recrystallization. This finally yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 20.1 gram (90% of theoretical) and the yield after five recrystallizations was 4.8 gram (18% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. Two analyses for nitrogen in gram 4.802 g., calculated: 4.800 g. the observed melting point of the pure compound was 102.5-103.5° C.

36. 1,4-bis-(2-oxyl)-1,4-bis-oxo-benzene) pyrenequinone

Empirical formula



Structural formula



molecular weight	468.52
boiling point	333.0-333.5° C.
yield of crude product	44g
yield after five recrystallizations	34g
analysis	found calculated
nitrogen	6.20% 6.07%
solubilities	
ether	insoluble
chloroform	insoluble
dioxane	insoluble
benzene	fairly soluble
toluene (perchloric acid extract)	fairly soluble
dichloroethane	fairly soluble
dichlorobenzene	soluble

24. Preparation of 1,4-dia-(2-methyl-4'-nitro-phenyl) piperazine.

Four and three-tenths grams (0.03 mole) of anhydrous piperazine was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 24.7 grams (0.1 mole) of 2-methyl-4'-nitro-phenol was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the jar for ten days, whereupon a pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 50% ethanol and then with a large amount of water. It was dissolved and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 25.8 grams (70% of theoretical) and the yield after three recrystallizations was 9.8 grams (28% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. This analyzed for nitrogen 18 gave 4.89% N. Calculated: 4.88% N. The corrected melting point of the pure compound was 126.5-126.5° C.

14. 1,4-bis-(2-methyl-2-butyl-oxalate) diglycolate

Empirical Formula

$C_{24}H_{44}O_{10}$

Structural Formula



Molecular Weight

468.57

Boiling Point

228.5-229.5° C.

Heat of Combustion

779

Heat after three decarboxylations

697

Analysis

Found Calculated

Carbon

4.89% 4.89%

Solubility

Water

Insoluble

Alcohol

Insoluble

Ether

Insoluble

Benzene

Slightly soluble

Toluene (decarboxylating solvent)

Slightly soluble

Dioxane

Very soluble

Chloroform

Soluble

18. Preparation of 1,4-bis-(2-methylpropyl-oxirane) ethane:

Four and three-tenths grams (0.08 mole) of adipic acid and one and a half grams (0.08 mole) of sodium acetate were dissolved in a mixture amount of boiling toluene under reflux. A hot solution solution of 17.0 grams (0.1 mole) of 2-methylpropyl-oxirane was then added. The total volume of the reaction was about 100 ml. The solution was refluxed over a three foot air condenser and then allowed to stand in the ice box one day, then was a pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of dry ethanol and then with a large amount of ether. It was dried and then recrystallized for three days toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 8.4 grams (84% of theoretical) and the yield after the recrystallization was 7.8 grams (84% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. When analyzed for adipic acid gave 4.44% H, calculated 4.44%. The corrected melting point of the pure compound was 100.5-100.5° C.

14. 1,4-bis-(4-oxocyclohexyl)butane-2,3-dione (g) studies.

Empirical formula

$C_{12}H_{18}O_4$

Structural formula



molecular weight

234.27

refractive index

$1.460-1.461^D_{20}$

Yield of crude product

84%

Yield after the recrystallization

84%

analysis

found calculated

nitrogen

4.50% 4.54%

solubilities

water

insoluble

alcohol

insoluble

ether

insoluble

benzene

fairly soluble

nitrobenzene (strongly oxidizing solvent)

fairly soluble

chloroform

fairly soluble

dichloroacetone

fairly soluble

15. Preparation of 4,4'-oxybis(1-methyl-2-chloro-aniline) preparation.

Four and three-tenths grams (0.08 mole) of aniline (purity value was checked in a standard amount of boiling toluene color value, a test which indicates 98.7 gram (0.1 mole) of 4-methyl-2-chloro-aniline was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a three foot reflux condenser and then allowed to stand in the ice box one day, whereupon a pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of water. It was dried and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 10.1 grams (50% of theoretical) and the yield after three recrystallizations was 6.7 grams (33% of theoretical). The product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. The analysis for nitrogen is given in Table II. Molecular weight 1.0000. The corrected melting point of the pure compound was 100.5-100.5° C.

71. 1,4-dioxane-(*p*-oxy)-1,4'-dioxane-styrene) copolymer.

Empirical formula

$C_{12}H_{16}O_2$

Structural formula



Molecular weight

208.27

Boiling point

200.0-205.0° C.

Yield of crude product

80%

Yield after three recrystallizations

80%

Analysis

Found (calculated)

C, %

67.8 (67.8)

Calculation

color

yellowish

flavor

pleasant

odor

characteristic

soluble

fairly soluble

in water (recrystallizing solvent)

fairly soluble

in benzene

fairly soluble

in chloroform

soluble

14. Preparation of 1,4-Di-(2-methyl-4'-tert-butylphenyl) pyrazole.

Four and three-tenths grams (0.028 mole) of anhydrous pyridine which was distilled in a vacuum vessel of boiling volume under reflux, 1.61 grams solution of 20.5 grams (0.1 mole) of 2-methyl-4'-tert-butylphenol was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a Dean-Stark for ten hours and then allowed to stand in the ice box one day, thereafter a pale yellow, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 95% ethanol and then with a large amount of ether. It was distilled and then recrystallized three times from toluene. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 0.8 grams (10% of theoretical) and the yield after three recrystallizations was 0.1 grams (1% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 4.87% N. Calculated: 4.87%. The corrected melting point of the pure compound was 133.5-135.5° C.

16. 1,1'-Bi-(2-allyl-2'-vinylbenzene) glycolate

empirical formula

$C_{20}H_{22}O_4$

structural formula



molecular weight

330.41

melting point

160.0-160.5° C.

yield of crude product

70g

yield after three recrystallizations

4g

analysis

found calculated

Carbon

6.77% 6.77%

solubility

water

insoluble

alcohol

insoluble

ether

partially

benzene

partly soluble

toluene (benzophenone solvent)

partly soluble

chloroform

soluble

dichloromethane

soluble

W. 1,4-Dioxane-(2,4-dimethyl-5-norbornene-2-thiocarbonyl) pyrazolone.

Four and three-tenths grams (0.05 mole) of adipic acid pyrazolone was dissolved in a mixture (100 ml) of boiling toluene under reflux. A hot toluene solution of 10.6 grams (0.1 mole) of 2,4-dimethyl-5-norbornene-2-thiocarbonyl was then added. The total volume of the solution was about 270 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the hot box for days, during which a pale yellow, crystalline compound separated out. The compound was filtered with care, washed with a small amount of 10% ethanol and then with a large amount of water. It was air-dried and then recrystallized by boiling with a large amount of toluene, cooling, and then filtering off the compound. This procedure was necessary since a suitable recrystallizing solvent could not be found. The substance, which in the solid state, is quite soluble in toluene. This process yielded a pale tan, crystalline compound. The yield of crude product was 10.6 grams (70% of theoretical) and the yield after purification was 11.6 grams (82% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. The analysis for nitrogen is given in Table I. The calculated nitrogen content of the pure compound was 18.6-18.6% N.

27. 3,4-Dihydro-2(1,4-dioxinyl)-2,2-dimethyl-1,3-dioxane (Dioxinyl)¹ polymerization.

Repeating formula



Structural formula



Monomer weight

166.20

Boiling point

204.5-205.5° C.

Yield of crude product

70%

Yield after purification

55%

Analysis

found calculated

Carbon

67.50% 67.70%

Hydrogen

Oxygen

Insoluble

Chloroform

Soluble

Water

Soluble

Ethanol

Slightly soluble

Diethyl ether

Slightly soluble

Acetone

Slightly soluble

Chloroform

Slightly soluble

III. Preparation of 1,4-bis-(2-methyl-oxazolone) piperazine.

Four and three-tenths grams (0.04 mole) of cuprous piperazine was dissolved in a minimum amount of boiling toluene under reflux. A hot toluene solution of 20.8 grams (0.1 mole) of 2-methyl-oxazolone was then added. The total volume of the solution was about half ml. The solution was refluxed over a Dean-Stark for ten hours and then allowed to stand in the ice box for days, changing the glass jar daily, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 70% ethanol and then with a large amount of water. It was alcoholized and then recrystallized three times from toluene. This yielded a pure, white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 12.8 grams (70% of theoretical) and the yield after three recrystallizations was 7.4 grams (45% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 5.33% N. Calculated: 5.48% N. The corrected melting point of the pure compound was 112.5-113.5° C.

III. 1,4-Bis-(1-methyl-phenyl) diphenyls.

Empirical formula

$C_{20}H_{16}$

Structural formula



Molecular weight

260.34

Melting point

116-116.5° C.

Yield of crude product

70g

Yield after three recrystallizations

40g

Analysis

Found: Calculated

C, 84.5%

84.5% 84.5%

Refractive index

n_D^{20}

1.5036

n_D^{25}

1.4976

n_D^{30}

1.4916

Refractive index

1.4916 (20°C)

Refractive index (recrystallizing solvent)

1.4916 (20°C)

Refractive index

1.4916

Refractive index

1.4916 (20°C)

22. Preparation of 1,4-Di-(2,4'-dinitrophenyl) piperazine

Over and over-bromide gram (2.18 mole) of adipic acid piperazine was dissolved in a solution amount of boiling toluene under reflux. A hot toluene solution of 22.8 gram (2.1 mole) of 2,4'-dinitrophenol was then added. The total volume of the solution was about 100 ml. The solution was refluxed over a free flame for ten hours and then allowed to stand in the lab for two days, through the solvent pure white, crystalline material separated out. The material was filtered with vacuum, washed with a small amount of 10% ethanol and then with a large amount of ether. It was air-dried and then recrystallized three times from toluene. This yielded a pure white, crystalline material. The crystals were washed with ether after the final recrystallization. The yield of crude product was 22.4 gram (70% of theoretical) and the yield after three recrystallizations was 9.8 gram (34% of theoretical). The purified product was dried in vacuum over concentrated sulfuric acid before being subjected to analysis. This analyzed for nitrogen 14 (pure 8.14%) N, calculated, 8.14% N. The corrected melting point of the pure compound was 122.5-124.5° C.

10. 1,4-dioxane-(1,4-dioxane)-dioxane polymerization

Structural formula



Structural formula



molecular weight

886.80

boiling point

209.8-210.2° C.

Heat of fusion (cal/g)

14.2

Heat after three isopropylations

14.2

soluble

good insoluble

chloroform

soluble insoluble

insoluble

water

insoluble

ether

insoluble

ether

insoluble

nitrobenzene

slightly soluble

nitrobenzene (crystallizing solvent)

slightly soluble

dioxane

soluble

dioxane

slightly soluble

B. Higher Members with Piperazine

1. Hexamethylenimine

A toluene solution of equimolar quantities of adipic acid piperazine and hexamethylenimine was refluxed for ten hours, and then allowed to stand in the ice box three days. The bright yellow crystals which separated out were recrystallized from alcohol, and mixed melting points proved them to be hexamethylenimine.

2. 2,4-dihydro-2H-pyridine (pipermaldehydamine).

A toluene solution of equimolar quantities of adipic acid piperazine and pipermaldehydamine was refluxed for ten hours, and then allowed to stand in the ice box ten days. The yellow crystals which separated out were recrystallized from alcohol three times, and mixed melting points proved them to be pipermaldehydamine.

3. 2-Mercaptoethane

A toluene solution of equimolar quantities of adipic acid piperazine and 2-mercaptoethane was refluxed for ten hours, and then allowed to stand in the ice box ten days. A black, heavy mass separated out, and was not further investigated.

4. Methyl cells.

Adipic acid piperazine and varying amounts of methyl cells were refluxed in various solvents such as benzene, toluene, and 10% alcohol, or still in alcohol a second. Varying amounts of adipic acid

ids were tried with the stochastic selection. In all cases, corrected algorithms appeared not, and was defeated by preparing the distance derivative and determining visual coding points with an arbitrary sample.

6. Structure constants of polyoxides.

1. Preparation of β -diatomic oxopolymers.

Polyoxides and oxo-oxides from (2.1) and (2.2) are obtained in about 10% yield, concentrated hydrogen peroxide. Oxopolymers and oxo-oxides from (2.1) and (2.2) are obtained in about 10% yield, and the solution refined over a fine filter for ten hours. It was then allowed to stand in the ice box for ten days, through the pure oxides, oxopolymers and oxo-oxides. The oxopolymers were filtered with water, washed with a small amount of hot water and then with a large amount of water. It was dried and then reprecipitated three times from concentrated hydrogen peroxide. This yielded a pure oxide, oxopolymers and oxo-oxides. The oxopolymers were washed with ether after the final reprecipitation. The yield of oxo product was 0.1 gram (10% of theoretical) and the yield after three reprecipitations was 0.1 gram (10% of theoretical). The purified product was dried in a vacuum oven at 100°C. and before being subjected to analysis. This analysis for nitrogen is given in Table I. (calculated: 4.70% N). The theoretical melting point of the pure oxopolymers was 100°C.

4. 4-Dichloroazobenzene

Empirical Formula



Molecular Formula



Molecular Weight

254.17

Melting Point

80.5-81.5° C.

Yield of crude product

80%

Yield after three recrystallizations

60%

Analysis

Found Calculated

Carbon

63.80% 63.80%

Calculation

Water

Insoluble (see test)

Alcohol

Very soluble

Ether

Very soluble

Dipropyl ether

Partly soluble

Hexane (recrystallizing solvent)

Partly soluble

Toluene

Very soluble

Dichlorane

Very soluble

Acetic

Very soluble

Diethyl sul.

Soluble

4. Preparation of 4-(4-methyl-oxolan-2-yl) morpholine.

Poly-four and four-butyle grams (3.8 mole) of 4-methyl-oxolan-2-one was dissolved in about 200 ml. of boiling, commercial heptane in the kettle. Twenty-two and one-half grams (0.2 mole + 0.2 mole excess) of sodium acrylate was then added, and the solution refluxed over a few days for two hours. It was then allowed to stand in the jar for two days, through the pure white, crystalline compound separated with the majority was filtered with strainer, washed with a small amount of dry ethanol and then with a large amount of water. It was dried and then recrystallized three times from commercial heptane. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 46.8 grams (70% of theoretical) and the yield after three recrystallizations was 41.8 gram (60% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 4.70% calculated 4.85% N. The corrected melting point of the pure compound was 41.5-42.5° C.

11. 4-(1-hydroxy-2-naphthyl) propylamine

Empirical formula



Structural formula



Molecular weight

253.35

Boiling point

211.0-213.0° C.

Yield of crude product

84g

Yield after three recrystallizations

84g

Analysis

Found: calculated

C, 80.9%

81.7% 80.9%

Solubilities

Water

Insoluble (see test)

Alcohol

Very soluble

Ether

Very soluble

Diethyl ether

Very soluble

Hexane (recrystallizing solvent)

Partly soluble

Toluene

Very soluble

Dichloroform

Very soluble

Acetone

Very soluble

Diethyl sul

soluble

B. Preparation of 4-(1-chloro-ethyl) morpholine.

Yield and melting point (lit. value) of 4-chloro-ethylamine was described as about 100 g., of boiling, commercial hydroxyethylamine. Yield and melting point (lit. value = 4.00) of nitrogen morpholine was also noted, and the solution refluxed over a few days for ten hours. It was allowed to stand in the ice box for days, then open the alcohol pure white, crystalline compound separated out. The compound was filtered with suction, washed with a small amount of 70% ethanol and then with a large amount of water. It was dried and then recrystallized three times from commercial hydroxyethylamine. This yielded a pure white, crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 11.0 grams (74% of theoretical) and the yield after three recrystallizations was 11.0 grams (77% of theoretical). The purified product was dried in vacuo over phosphorus pentoxide and before being subjected to analysis. When analyzed for nitrogen it gave 4.00% N. calculated: 4.00% N. The reported melting point of the pure compound was 33-34.0° C.

II. 4-(p-tolylamino)azobenzene

Empirical formula



Structural formula



Molecular weight

209.28

Boiling point

99.5-100.5° C.

Yield of crude product

70g

Yield after three recrystallizations

47g

Analysis

Found (calculated)

Carbon

81.8% (81.8%)

Solubility

Water

Insoluble (ins. hot)

Methanol

Soluble

Ether

Very soluble

Drypyrid ether

Very soluble

Dioxane (recrystallizing solvent)

Fairly soluble

Chloroform

Very soluble

Dichloroethane

Very soluble

Acetone

Very soluble

Methyl alcohol

Soluble

4. 4-(3'-deoxy-2'-thio) cytosine

Empirical formula



Structural formula



Molecular weight

244.28

Melting point

262-263.5° C.

Yield of crude product

60g

Yield after three recrystallizations

17g

Analysis

Found: calculated

C, 49.5%

47.9% 47.9%

H, 4.9%

N, 23.6%

23.6% (theor. 23.6%)

S, 12.0%

12.0%

OH, 0%

0%

2'-deoxyribose

0%

Uracil (recrystallized solvent)

100% soluble

Thiouracil

very soluble

Thiothymine

very soluble

Adenine

very soluble

Uridine 5P

very soluble

5. Preparation of 2-(1'-methyl-ethoxycarbonyl) acrylonitrile.

Twenty-two and ten-fourths grams (71.1 mmole) of 2-methyl-but-3-ene was dissolved in about 100 ml. of boiling, concentrated hydrochloric acid. Thirteen and one-fourth grams (41.55 mmole + 4.15 mmole excess) of anhydrous acrylonitrile was then added, and the solution refluxed over a fire flame for ten hours. It was allowed to stand in the ice box ten days, during which almost pure white, acrylonitrile composed copolymer was formed. The copolymer was filtered with suction, washed with a small amount of 10% ethanol and then with a large amount of water. It was distilled and then reprecipitated three times from concentrated hydrochloric acid. This yielded a pure white, acrylonitrile copolymer. The copolymer was washed with ether after the final reprecipitation. The yield of white product was 29.1 grams (74% of theoretical) and the yield after three reprecipitations was 24.8 grams (74% of theoretical). The product yielded the data in table over concentrated sulfuric acid before being subjected to analysis. When analyzed for nitrogen it gave 4.0% N, calculated, 4.10% N. The corrected melting point of the pure copolymer was 27.5-28.5° C.

6. 6-(4'-methoxyphenyl) acrylonitrile.

Empirical formula

$C_{10}H_{11}NO_2$

Structural formula



Molecular weight

177.20

Boiling point

101.5-102.5° C.

Yield of crude product

14g

Yield after three recrystallizations

7g

Analysis

Found: calculated

C, 68.3%

67.8% 68.0%

Properties

Water

insoluble (dec. hot)

Methanol

soluble

Ether

very soluble

Dioxane¹ ether

very soluble

Hexane (recrystallizing solvent)

fairly soluble

Toluene

very soluble

Chloroform

very soluble

Acetone

very soluble

Methyl cel

soluble

4. Preparation of 4,4'-[2,2-bis(4-chlorophenoxy)]-2,2'-biphenol.

Seven and seven-tenths grams (0.02 mole) of 2,2-bis(4-chlorophenoxy)propane dissolved in about 200 ml of boiling, concentrated hydrochloric acid. Sixteen and one-tenth grams (0.02 mole + 0.02 mole excess) of cuprous hydrochloride was then added, and the solution refluxed over a five liter jar for ten hours. It was then allowed to stand in the jar for about days, through the almost pure white, crystalline compound separated out, and the pale yellow oil which stayed suspended not solidified. The whole mass was filtered with suction, washed with a small amount of dry ethanol and then with a large amount of water. It was air-dried and then recrystallized six times from concentrated hydrochloric acid. The product is almost pure white (very pale tan), crystalline compound. The crystals were washed with ether after the final recrystallization. The yield of crude product was 22.1 grams (90% of theoretical) and the yield after six recrystallizations was 22.1 grams (80% of theoretical). The purified product was dried in vacuo over concentrated sulfuric acid before being subjected to analysis. This material for analysis is pure 4,4'-[2,2-bis(4-chlorophenoxy)]-2,2'-biphenol. The corrected boiling point of the pure compound was 225.5-226.5° C.

6. 4,4'-(2,2,6,6-tetramethyl-1,3-bisoxepane)bisoxepane

isolated product

92/90/90%

theoretical product



solubility (mg/ml)

solubility

solubility (mg/ml)

solubility (mg/ml)

Yield of crude product

80%

Yield after the recrystallization

80%

analysis

found calculated

C, 66.00%

65.80% 65.80%

calculated

water

insoluble (non. sol.)

ethanol

soluble

ether

soluble

propyl ether

soluble

hexane (recrystallizing solvent)

fairly soluble

toluene

very soluble

dichloroethane

very soluble

acetone

very soluble

ethyl acetate

very soluble

3. Negative reactions with morpholine

1. 4-nitrophenyl-bromo-stalones.

A commercial heptane solution of 0.01 mole of 4-nitrophenyl-bromo-stalones and 0.005 mole of morpholine was refluxed for ten hours, and then allowed to stand in the ice box ten days. The yellow crystals which separated out were recrystallized from commercial heptane, and mixed melting points proved them to be the unreacted stalones.

2. 4-nitrophenyl-ethyl-bromo-stalones.

A commercial heptane solution of 0.01 mole of 4-nitrophenyl-ethyl-bromo-stalones and 0.1 mole of morpholine was refluxed for ten hours, and then allowed to stand in the ice box ten days. The yellow crystals which separated out were recrystallized from commercial heptane, and mixed melting points proved them to be the unreacted stalones.

3. 4-nitrophenyl-ethyl-bromo-stalones.

A commercial heptane solution of 0.01 mole of 4-nitrophenyl-ethyl-bromo-stalones and 0.005 mole of morpholine was refluxed for ten hours, and then allowed to stand in the ice box ten days. The yellow crystals which separated out were recrystallized from commercial heptane, and mixed melting points proved them to be the unreacted stalones.

4. Dimethylacetophenone.

A commercial heptane solution of 0.01 mole of dimethylacetophenone and 0.1 mole of morpholine was refluxed for ten hours, and then al-

level to stand in the low box for days. The bright yellow crystals which separated out were recrystallized from 95% ethanol, and mixed melting points proved them to be the unreacted monomaleic anhydride.

6. 4-Methyl-2-oxocyclohexanone,

A commercial heptane solution of 5.00 mole of 4-methyl-2-oxocyclohexanone and 5.000 mole of acrylonitrile was refluxed for ten hours, and then allowed to stand in the low box for days. The yellow crystals which separated out were recrystallized from commercial heptane, and mixed melting points proved them to be the unreacted diene.

7. 4,4'-Dinitrochlorobenzene,

A commercial heptane solution of 5.1 mole of 4,4'-dinitrochlorobenzene and 5.10 mole of acrylonitrile was refluxed for ten hours, and then allowed to stand in the low box for days. The almost pure white crystals which separated out were recrystallized from hexane from commercial heptane, yielding pure yellow crystals. Mixed melting points proved them to be the diene, apparently the addition compound is too unstable to be isolated in the pure state.

8. 4,4'-Dichlorobenzene,

A commercial heptane solution of 5.000 mole of 4,4'-dichlorobenzene and 5.1 mole of acrylonitrile was refluxed for ten hours, and then allowed to stand in the low box for days. The almost pure white crystals which separated out were recrystallized from hexane from commercial heptane, yielding pure yellow crystals. Mixed melting points proved them to be the diene, resulting from the unstable addition.

PREPARATION OF ISOMERISMS

1. *Diastere; trans-isomerism.*

The known product, melting point $82-83^{\circ}$ C. was used in the present.

trans-isomerism is prepared readily by dissolving equimolar quantities of benzaldehyde and acetophenone in 200 c.c. and adding 100 grams of a 10% aqueous solution of sodium hydroxide per mole of aldehyde (200 grams). The compound may be recrystallized from 200 c.c. of alcohol. A detailed procedure is given by Gilman²⁷.

2. *2-ethyl-diastere.*

Twenty ml. (10.0 grams (0.8 mole)) of *n*-ethyl-benzaldehyde and 10.0 grams (0.8 mole) of acetophenone were dissolved in about 700 ml. of pure 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, through a 14 mesh gauze which almost immediately. It was allowed to stand in the lab for one day, filtered with caution, washed with a large amount of water, and dried. The compound was recrystallized three times from benzene, yielding pale yellow crystals. The yield after three recrystallizations was 10.8 grams (95% of theoretical). The corrected melting point of the pure product was $44.5-45.5^{\circ}$ C. The melting point reported in the literature²⁸ is 44° ; $44-45^{\circ}$ C.

4. 2-Dimethylsiloxane.

Twenty-six and eight-tenths grams (2.2 mole) of methoxybenzohydroxy and 48.0 grams (2.2 mole) of methoxyphenol were dissolved in about 100 ml. of 95% ethanol. Thirty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly and then allowed to stand in the lab box one day, changing the pale yellow, crystalline suspension separated out. The crystals were filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized three times from 95% ethanol, yielding pale yellow crystals. The yield after three recrystallizations was 37.0 grams (70% of theoretical). The corrected melting point of the pure product was 40.0-40.1°C.

5. 2'-Dimethylsiloxane.

Twenty-one and two-tenths grams (2.2 mole) of benzohydroxy and 48.0 grams (2.2 mole) of p-methoxyphenol were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, changing to the pale yellow suspension separated out almost immediately. It was allowed to stand in the lab box one day, filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized twice from 95% ethanol, yielding pale yellow crystals. The yield after the recrystallizations was 44.0 grams (80% of theoretical). The corrected melting point of the pure product was 34.0-34.1°C.

2'-Dimethylsiloxane has been prepared by adding a solution

solution of the samples and before to an ethanol solution of sodium hydroxide¹⁰. Therefore in this paper we used as a recrystallizing solvent. This research has shown that the compound in fact exhibits in these solvents to melt their own function. The above references reports the melting point to be 111^o.

3. Isobutyl-ethanol.

Thirty-five and one-tenth grams (3.50 mole) of p-chloro-benzonitrile and 31.5 grams (3.50 mole) of isobutylalcohol were dissolved in about 100 ml. of 95% ethanol. Twenty-five grams of a 30% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, changes in volume could almost immediately. It was allowed to stand in the lab for one day, filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from 95% ethanol, yielding pale yellow crystals. The yield after three recrystallizations was 23.5 grams (68% of theoretical). The observed melting point of the pure compound was 113.5-113.6^o is the melting point reported in the literature¹⁰ is 108-109^o g.

4. 4,4'-Dichlorobenzene.

Twenty-five grams (0.5 mole) of p-chlorobenzonitrile and 31.5 grams (3.5 mole) of p-chlorobenzonitrile were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 30% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, changes in pale yellow, crystalline compound separated out almost immediately. It

was allowed to stand in the ice box one day, filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from ethyl acetate, yielding pale yellow needles. The yield after three recrystallizations was 66.7 grams (77% of theoretical). The corrected melting point of the pure compound was 188.4-189.4° C. The melting point reported in the literature²⁴ is 188-189° C.

V. 4-*trans*-isomers.

Twenty-one and ten-tenths grams (5.0 mols) of benzaldehyde and 55.0 grams (5.0 mols) of *p*-bromobenzene were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 5% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, decanting the pale yellow, crystalline compound separated and dried immediately. It was allowed to stand in the ice box one day, filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from 95% ethanol, yielding pale yellow crystals. The yield after three recrystallizations was 48.5 grams (70% of theoretical). The corrected melting point of the pure compound was 204.2-205.4° C. The melting points recorded in the literature are melting. Hottel, et al.,²⁵ report it to be 204-205° C.; Hottel and Dreyer²⁶ report it as 205-204° C.

b. 4-*trans*-isomers.

Twenty grams (5.0 mols) of *p*-chlorobenzaldehyde and 55.0 grams (5.0

ml.) of anhydrous was dissolved in about 50 ml. of 95% ethanol. Thirty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, and then allowed to stand in the ice box one day, whereupon the pale yellow, crystalline compound separated out. It was filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized once from commercial heptane, yielding pale yellow crystals. The yield after one recrystallization was 34.8 grams (70% of theoretical). The corrected melting point of the pure compound was 34.5-35.5° C. The melting point reported in the literature¹⁰ is 34.5° C.

5. 2'-Methyl-chloran.

Eighty-three grams (0.8 mole) of benzaldehyde and 45.8 grams (0.8 mole) of *p*-nitro-*o*-chlorophenol were dissolved in about 100 ml. of 95% ethanol. Fifty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was thoroughly stirred and then allowed to stand in the ice box one day, whereupon the pale yellow crystalline compound separated out. It was filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized three times from diethyl ether, yielding pale yellow crystals. The yield after three recrystallizations was 45.8 grams (100% of theoretical). The corrected melting point of the pure compound was 71.5-73.5° C. The melting point reported in the literature is 72-73° C.¹⁰

10. 4-Hydroxy-2-butanone.

Twenty-eight grams (0.2 mole) of acetylacetone and 20.0 gram (0.2 mole) of acetylbenzene were dissolved in about 100 ml. of 95% ethanol. Fifty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly and then allowed to stand in the ice box one day, through the pale yellow, crystalline compound separated out. It was filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from 95% ethanol, yielding pale yellow crystals. The yield after three recrystallizations was 17.0 gram (55% of theoretical). The corrected melting point of the pure compound was $74.5-74.6^{\circ}$ C. The melting point reported in the literature²² is $77-78^{\circ}$ C.

11. 4,4'-Diacetyl-2-butanone.

Twenty-four grams (0.2 mole) of p-toluidiacetyl and 20.0 gram (0.2 mole) of p-acetyl-acetylbenzene were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, through the pale yellow, crystalline compound separated out almost immediately. It was allowed to stand in the ice box one day, filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from 95% ethanol, yielding pale yellow crystals. The yield after three recrystallizations was 11.0 gram (45% of theoretical). The corrected melting point of the pure compound was $117.5-118.0^{\circ}$ C.

12. 4-hydroxy-4'-methyl-stilbene.

Twenty-seven and ten-tenths grams (3.2 mols) of acetylacetyl and 26.6 grams (3.2 mols) of p-methyl-acetophenone were dissolved in about 50 ml. of 95% ethanol. Twenty grams of a 5% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly and then allowed to stand in the ice box one day, during the pale yellow, crystalline compound separated out. It was filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized twice from 95% ethanol, yielding pale yellow crystals. The yield after two recrystallizations was 22.6 grams (88% of theoretical). The corrected melting point of the pure compound was 55.5-57.0° C.

13. Hexamethylacetophenone.

Twenty-eight and eight-tenths grams (3.27 mols) of acetone acetyl and 44.4 grams (3.27 mols) of acetophenone were dissolved in about 50 ml. of 95% ethanol. Twenty grams of a 5% aqueous solution of sodium hydroxide was then added. The solution was thoroughly stirred and then allowed to stand in the ice box one day, during the bright yellow, crystalline compound separated out. It was filtered with suction, washed with a large amount of water and air-dried. The compound was recrystallized three times from 95% ethanol, yielding bright yellow crystals. The yield after three recrystallizations was 26.4 grams (94% of theoretical). The corrected melting point of the pure product was 55.5-56.5° C. The melting point reported in the literature²⁰ is 55-57° C.

14. 4-bromophenyl-2-bromo-ethanes.

Twenty-four and ten-tenths grams (9.8 mols) of succinimide and 25.0 grams (9.8 mols) of p-bromotoluene were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, and the pale yellow, crystalline compound separated out almost immediately. The compound was allowed to stand in the ice box one day, then filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized once from a large amount of concentrated hydroxide, yielding pale yellow crystals. The yield after one recrystallization was 19.8 grams (50% of theoretical). The corrected melting point of the pure compound was 141.5-142.5° C.

15. 4-bromophenyl-2-bromo-ethanes.

Twenty-four grams (9.8 mols) of p-toluenesulfonic acid and 25.0 grams (9.8 mols) of p-bromotoluene were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly and then allowed to stand in the ice box one day, whereupon the pale yellow, crystalline compound separated out. It was filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized once from a large amount of concentrated hydroxide, yielding pale yellow crystals. The yield after one recrystallization was 19.8 grams (50% of theoretical). The corrected melting point of the pure compound was 140.5-141.5° C.

16. 1-Hydroxy-2-naphthol-sulfonic.

Twenty-four grams (0.1 mole) of *o*-toluidinyloxy and 24.2 grams (0.2 mole) of *p*-chlorobenzenesulfonic were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, evaporated to pale yellow, crystalline compound separated out almost immediately. The compound was allowed to stand in the ice box one day, then filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from a small amount of concentrated hydrochloric acid, yielding pale yellow crystals. The yield after three recrystallizations was 17.1 grams (70% of theoretical). The corrected melting point of the pure compound was 154.8-155.0° C.

17. 1-Hydroxy-2-naphthol-sulfonic.

Twenty-seven and two-tenths grams (0.1 mole) of *o*-toluidinyloxy and 24.2 grams (0.2 mole) of *p*-chlorobenzenesulfonic were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, evaporated to pale yellow, crystalline compound separated out almost immediately. The compound was allowed to stand in the ice box one day, then filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from a large amount of concentrated hydrochloric acid, yielding pale yellow, fairly massive. The yield after three recrystallizations was 21.7 grams (84% of theoretical). The corrected melting point of the pure compound was

III.5-III.6² g.

10. 4-Hydroxy-2,6-dimethylbenzoic acid.

Twenty-four grams (0.4 mole) of p-toluenesulfonic acid and 51.0 grams (0.4 mole) of p-toluenesulfonamide were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, turning the pale yellow, crystalline compound separated out almost immediately. The compound was allowed to stand in the lab for one day, then filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized three times from a large amount of commercial kerosene, yielding pale yellow needles. The yield after the recrystallization was 51.0 grams (70% of theoretical). The corrected melting point of the pure compound was III.5-III.6² g.

11. 4-Hydroxy-2,6-dimethylbenzoic acid.

Twenty-four grams (0.4 mole) of p-toluenesulfonic acid and 51.0 grams (0.4 mole) of p-toluenesulfonamide were dissolved in about 100 ml. of 95% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, turning the pale yellow, crystalline compound separated out almost immediately. The compound was allowed to stand in the lab for one day, then filtered with suction, washed with a large amount of water, and dried. The compound was recrystallized three times from commercial kerosene, yielding pale yellow needles. The yield after three recrystallizations

was 44.4 grams (100% of theoretical). The corrected melting point of the pure compound was 114.5-114.6° C.

10. 2,4'-Dinitro-1-naphthol.

Twenty-five grams (1.44 mole) of *n*-butylaldehyde and 44.4 grams (1.44 mole) of *p*-nitro-1-naphthol were dissolved in about 100 ml. of 80% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, whereupon the compound separated out almost immediately in the form of an oil. The solution was allowed to stand in the ice box one day and the oil changed to pale yellow crystals. The compound was then filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized three times from a small amount of concentrated hydrochloric acid giving pale yellow crystals. The yield after three recrystallizations was 44.4 grams (100% of theoretical). The corrected melting point of the pure compound was 76.5-76.6° C.

11. 2-Nitro-1-naphthol.

Twenty-eight grams (1.44 mole) of *n*-butylaldehyde and 44.4 grams (1.44 mole) of 1-naphthol were dissolved in about 100 ml. of 80% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, whereupon the compound separated out almost immediately in the form of an oil. The solution was allowed to stand in the ice box one day and the oil changed to pale yellow crystals. The compound was then filtered with suction,

mixed with a large amount of water, and stirred. The compound was recrystallized three times from a small amount of concentrated hydrofluoric acid. The yield after three recrystallizations was 0.6 gram (60% of theoretical). The corrected melting point of the pure compound was 111.5-112.0° C.

15. 1,4-Dihydroxy-2-methyl-2-propyl-3-oxocyclohexane.

Twenty grams (0.1 mole) of pyruvic acid and 40.0 grams (0.2 mole) of cyclohexane were dissolved in about 100 ml. of 80% ethanol. Thirty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, changed the color, crystalline material separated out almost immediately, the solution was allowed to stand in the lab for one day, and the compound was filtered with water, washed with a large amount of water, and distilled. The compound was recrystallized one time from 80% ethanol, yielding yellow crystals. The yield after one recrystallization was 10.0 grams (50% of theoretical). The corrected melting point of the pure compound was 121.0-121.5° C. The melting point reported in the literature²⁷ is 120° C.

16. 1,4-Dihydroxy-2-methyl-2-propyl-3-oxocyclohexane.

Twenty grams (0.1 mole) of pyruvic acid and 40.0 grams (0.2 mole) of p-bromocyclohexane were dissolved in about 100 ml. of 80% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was thoroughly stirred, changed the

gale polim, crystalline compound separated out almost immediately. The solution was allowed to stand in the ice box one day, and the supernatant was filtered with suction, washed with a large amount of water, and air-dried. The compound was recrystallized once more from alcohol four times of commercial hydroxyl, yielding pale yellow crystals. The yield after one recrystallization was 35.4 grams (80% of theoretical). The corrected melting point of the pure compound was 133.5-134.5° C.

14. Diethyl-maleate.

Thirty and five-tenths grams (35.5 gms.) of *n*-butyl-maleate hydroxide and 35.5 grams (35.5 mols) of methoprene were dissolved in about 100 ml. of 10% ethanol. Twenty grams of a 10% aqueous solution of sodium hydroxide was then added. The solution was stirred thoroughly, through a cork lower magnetic separator and almost immediately. The solution was allowed to stand in the ice box one day, and then filtered with suction. The diethyl-maleate crystals were washed with water, air-dried, and recrystallized three times from alcohol; the alcohol solution was boiled with "sugar" during the first recrystallization. This yielded fluffy, orange crystals. The yield after three recrystallizations was 35.5 grams (80% of theoretical). The corrected melting point of the pure compound was 133.5-134.5° C.

15. Diethyl-maleate.

The procedure used in that listed as "Diethyl-maleate"¹⁰.

A mixture of 100 grams (1 mole) of benzothiazole and 50 grams (0.5 mole) of sodium is added, with mechanical stirring, to a cooled solution of 100 grams of sodium hydroxide in 1000 ml. of water and 500 ml. of 95% ethanol. The mixture is stirred for about one hour, and the mass is then filtered with suction and washed with a large amount of water. The product was air-dried and then recrystallized from twice from ethyl acetate, yielding bright yellow crystals. The yield after two recrystallizations was 60.0 grams (60% of theoretical). The corrected melting point of the pure compound was 120.8-121.4° C.

SUMMARY

1. The following new addition compounds of pyrazoline with aldehydes

	Page
(1.) 1,4-Di-(2-thioure)-pyrazoline	25
(2.) 1,4-Di-(2-thioure)-aldehyde pyrazoline	26
(3.) 1,4-Di-(2-thioure)-aldehyde a) pyrazoline	27
(4.) 1,4-Di-(2 ¹ -thioure)-aldehyde) pyrazoline	28
(5.) 1,4-Di-(2-thioure)-aldehyde a) pyrazoline	21
(6.) 1,4-Di-(2,4 ¹ -thioure)-aldehyde) pyrazoline	29
(7.) 1,4-Di-(2 ¹ -thioure)-aldehyde a) pyrazoline	29
(8.) 1,4-Di-(2-methyl)-aldehyde a) pyrazoline	27
(9.) 1,4-Di-(2 ¹ -methyl)-aldehyde a) pyrazoline	28
(10.) 1,4-Di-(2-methyl-g)-aldehyde) pyrazoline	32
(11.) 1,4-Di-(2,4 ¹ -methyl)-aldehyde) pyrazoline	33
(12.) 1,4-Di-(2-methyl-g ¹ -methyl)-aldehyde) pyrazoline	33
(13.) 1,4-Di-(2-methyl-g ¹ -thioure)-aldehyde) pyrazoline	37
(14.) 1,4-Di-(2-methyl-1,4 ¹ -thioure)-aldehyde) pyrazoline	38
(15.) 1,4-Di-(2-methyl-1,4 ¹ -thioure)-aldehyde a) pyrazoline	39
(16.) 1,4-Di-(2-methyl-g ¹ -thioure)-aldehyde) pyrazoline	39
(17.) 1,4-Di-(2-methyl-1,4 ¹ -thioure)-aldehyde a) pyrazoline	44
(18.) 1,4-Di-(2-methyl-1,4 ¹ -thioure)-aldehyde) pyrazoline	37
(19.) 1,4-Di-(2,4 ¹ -methyl-1,4 ¹ -g ¹ -thioure)-aldehyde) pyrazoline	40
(20.) 1,4-Di-(2-methyl)-aldehyde) pyrazoline	44
(21.) 1,4-Di-(2,4 ¹ -methyl)-aldehyde) pyrazoline	35

6. The following alpha, beta-unsaturated ketones were not successfully reacted with pyridine under conditions which were successful with the foregoing compounds:

	Page
(1.) Diacetylphenone	68
(2.) 1,4-diacetylene-2,5-py-dione; Diphenylacetylphenone	68
(3.) 2,4-diacetylene	68
(4.) Acetyl anhydride	68

7. The following are additional compounds of acrylonitrile with alpha, beta-unsaturated ketones have been prepared and studied:

(1.) 4-stilbene acrylonitrile	67
(2.) 4-(4-methyl-stilbene) acrylonitrile	68
(3.) 4-(4-chloro-stilbene) acrylonitrile	72
(4.) 4-(4'-bromo-stilbene) acrylonitrile	71
(5.) 4-(4'-methyl-stilbene) acrylonitrile	71
(6.) 4,4'-[2,2-bis(4-oxocyclohexyl)]-5,5'-diacrylonitrile	77

8. The following alpha, beta-unsaturated ketones were not successfully reacted with acrylonitrile, or could not be isolated, under conditions which were successful with the foregoing compounds:

(1.) 4-methyl-4'-bromo-stilbene	70
(2.) 4-methyl-4'-chloro-stilbene	70
(3.) 4-methyl-4'-bromo-stilbene	70
(4.) Diacetylphenone	70
(5.) 4-methyl-4'-chloro-stilbene	70
(6.) 4,4'-[2,2-bis(4'-chloro-1-cyclohexyl)]-5,5'-diacrylonitrile	70

(7.)	$4_2 \cdot 2^2$ -graph Latin squares	88
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8. The following cyclic, semi-structured Latin Squares have been prepared and converted physical form (approximately):

(1.)	Orthogonal Design Construction	92
(2.)	0-22 Latin Square	93
(3.)	0-26 Latin Square	93
(4.)	4 ² -orthogonal Latin Square	93
(5.)	4-orthogonal Latin Square	93
(6.)	$4_2 \cdot 4^2$ -orthogonal Latin Square	93
(7.)	4^2 -orthogonal Latin Square	94
(8.)	4-cyclic Latin Square	94
(9.)	4^2 -cyclic Latin Square	94
(10.)	4-cyclic square Latin Square	94
(11.)	$4_2 \cdot 4^2$ -cyclic Latin Square	94
(12.)	4-cyclic square of 4-cyclic Latin Square	97
(13.)	0-22 Latin Square	97
(14.)	4-cyclic square of 4 ² -orthogonal Latin Square	98
(15.)	4-cyclic square of 0-26 orthogonal Latin Square	98
(16.)	4-cyclic square of $4_2 \cdot 4^2$ -orthogonal Latin Square	98
(17.)	4-cyclic square of 4^2 -orthogonal Latin Square	98
(18.)	4-cyclic square of 0-26 orthogonal Latin Square	98
(19.)	0-26 cyclic Latin Square	98
(20.)	$4_2 \cdot 4^2$ -cyclic Latin Square	98
(21.)	0-cyclic Latin Square	98

(111.)	S_1 -Hilf (2.2.1)–(2.2.2)–(2.2.3)	Page 88
(112.)	S_1 -Hilf (2.2.1)–(2.2.2)–(2.2.3)–(2.2.4)	88
(113.)	S_1 -Hilf (2.2.1)–(2.2.2)	89
(114.)	S_1 -Hilf (2.2.1)–(2.2.2)	89

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BIOGRAPHY

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PROPOSAL REPORT

This Dissertation was prepared under the supervision of the Chairman of the candidate's Department Committee, and has been approved by all members of the Committee. It was submitted to the Faculty and was approved as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

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